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(54) PRODUCTION OF HIGH-PURITY COPPER INGOT

(57)Abstract:

PURPOSE: To produce a high-purity copper ingot contg. Fe and Si at extremely low ratios by using a carbon crucible, nozzle and casting mold.

CONSTITUTION: This method comprises producing the high-purity copper ingot by melting a high-purity copper raw material, such as electric copper, by using the carbon crucible and passing the resulted high-purity copper melt through the carbon nozzle into the carbon casting mold, thereby casting the ingot. The carbon crucible, the carbon nozzle and the carbon casting mold are previously heat treated under a condition of holding at 800 to 1120°C in a non-oxidizing atmosphere before the high-purity copper raw material is melted and cast. The high-purity copper ingot is produced by using the heat treated carbon crucible, the carbon nozzle and the carbon casting mold.

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[Claim(s)]

[Claim 1] In the method of dissolving a high grade copper raw material using the crucible made from carbon, casting the obtained high grade copper molten metal to the mold made from carbon through the nozzle made from carbon, and manufacturing a high grade copper ingot Before dissolving and casting the above-mentioned high grade copper raw material, beforehand the above-mentioned crucible made from carbon, the nozzle made from carbon, and the mold made from carbon The inside of a non-oxidizing atmosphere, Temperature: The manufacturing method of the high grade copper ingot characterized by heat-treating [800-1120-degree C] the conditions of maintenance, and manufacturing a high grade copper ingot using this heat-treated crucible made from carbon, the nozzle made from carbon, and the mold made from carbon.

[Claim 2] In the method of dissolving a high grade copper raw material using the crucible made from carbon, casting the obtained high grade copper molten metal to the mold made from high purity alumina through the nozzle made from high purity alumina, and manufacturing a high grade copper ingot The manufacturing method of the high grade copper ingot characterized by giving the inside of a non-oxidizing atmosphere to the above-mentioned crucible made from carbon, heat-treating the conditions of maintenance at temperature:800-1120 degree C beforehand, and dissolving the above-mentioned high grade copper raw material using this heat-treated crucible made from carbon before dissolving the above-mentioned high grade copper raw material.

[Claim 3] In the method of dissolving a high grade copper raw material using the crucible made from carbon, casting the obtained high grade copper molten metal to the mold made from high purity alumina through the nozzle made from carbon, and manufacturing a high grade copper ingot Before dissolving and casting the above-mentioned high grade copper raw material, beforehand the above-mentioned crucible made from carbon, and the nozzle made from carbon The inside of a non-oxidizing atmosphere, Temperature: The manufacturing method of the high grade copper ingot characterized by heat-treating [800-1120-degree C] the conditions of maintenance, and carrying out dissolution casting using this crucible made from carbon

and nozzle made from carbon that were heat-treated.

[Claim 4] In the method of dissolving a high grade copper raw material using the crucible made from carbon, casting the obtained high grade copper molten metal to the mold made from high grade carbon through the nozzle made from high purity alumina, and manufacturing a high grade copper ingot Before dissolving and casting the above-mentioned high grade copper raw material, beforehand the above-mentioned crucible made from carbon, and the mold made from carbon The inside of a non-oxidizing atmosphere, Temperature: The manufacturing method of the high grade copper ingot characterized by heat-treating [800-1120-degree C] the conditions of maintenance, and carrying out dissolution casting using this crucible made from carbon and mold made from carbon that were heat-treated.

[Claim 5] The heat treatment temperature of the above-mentioned crucible made from carbon, the nozzle made from carbon, and the mold made from carbon is a manufacturing method of a high grade copper ingot Claim 1, 2 and 3, or given in four characterized by being within the limits of 900-1050 degrees C.

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the manufacturing method of the high grade copper ingot for manufacturing high grade copper wire material, such as a bonding wire of the electric wire for audios, and a semiconductor device, and a superconductivity stabilizer.

[0002]

[Description of the Prior Art] Generally the high grade copper wire more than 99.9999 weight % (henceforth 6N) is used for the bonding wire of the electric wire for audios, and a semiconductor device, and the superconductivity stabilizer.

[0003] The high grade copper wire beyond these 6N manufactured the high grade copper ingot beyond 6N first, it rolled this high grade copper ingot, repeated wire drawing and annealing, and was manufacturing the high grade copper wire which finally has a predetermined narrow diameter by wire drawing.

[0004] The high grade copper ingot beyond 6N is conventionally manufactured by casting a vacuum or the molten metal obtained by carrying out the inert gas atmosphere dissolution to the mold made from high purity alumina through the nozzle made from high purity alumina in the crucible made from high purity alumina for high grade electrolytic copper beyond 6N.

[0005] However, the high grade copper ingot manufactured using the crucible made from high purity alumina, the nozzle made from high purity alumina, and the mold made from high purity alumina is although the purity of the almost same grade as electrolytic copper can be maintained, A crucible, a nozzle, a mold, etc. made from high purity alumina broke easily, and it was easy to carry out them with **, and they had become what also has the expensive price of the high grade copper wire which in the top is obtained since it is expensive.

[0006] Therefore, high grade electrolytic copper was usually dissolved using the commercial crucible made from high grade carbon, and the high grade copper ingot was manufactured using the nozzle made from high grade carbon and the mold made from carbon of marketing of the obtained molten metal.

[0007]

[Problem(s) to be Solved by the Invention] However, if dissolution casting of the electrolytic copper of a high grade is carried out and a high grade ingot is manufactured using the crucible made from carbon, the nozzle made from carbon, and the mold made from carbon (these are hereafter named generically the furnace material made from carbon) which are called commercial high grade Fe and Si which are contained in the furnace material made from carbon are eluted, and Fe and Si are 0.5 ppm in penetration and the obtained high grade ingot in melted copper, respectively. It was unavoidable that a grade is contained.

[0008] The content of Fe and Si which are contained in the obtained high grade copper ingot even if this dissolves high grade electrolytic copper to the extent that Fe and Si cannot detect in a mass spectrograph as a raw material is 0.5 ppm, respectively. It is checked also from being contained above.

[0009] Fe and Si are 0.5 ppm to high grade copper wire material, respectively. if contained above a residual resistivity ratio (the ratio of electric resistance rho298K in a room temperature to electric resistance rho4.2K at liquid helium temperature -- [it defines as rho298 K/rho 4.2K, and]) the following and RRR -- describing -- [the development of a noise / increase rapidly and] if it is made to fall and the low high grade wire rod of this RRR is used for the electric wire for audios Even if it uses it as a superconductivity stabilizer, the electric resistance under very low temperature serves as size, and a desired effect is not acquired, but even if it uses it as a bonding wire of a semiconductor device, an adhesive property worsens.

[0010] Therefore, a certain thing is [RRR / 10,000 or more] required for the wire rod which can be used as the electric wire for audios, a superconductivity stabilizer, a bonding wire, etc. In order for RRR to carry out to 10,000 or more, it is more than 6N, and the content of Fe and Si is 0.1 ppm, respectively. from the place where it is required to be the following The content of Fe and Si is 0.1 ppm, respectively. The technical development which can manufacture the following high pure copper ingots inexpensive using the furnace material made from carbon was called for.

[0011]

[Means for Solving the Problem] Then, this invention person etc. uses the furnace material made from carbon, and the content of Fe and Si is 0.1 ppm, respectively. The result of having inquired in order to manufacture the following high grade copper ingots, (a) It is high grade electrolytic copper beforehand The inside of a non-oxidizing atmosphere, and temperature:800-1120 degree C (preferably) If the furnace material made from carbon is heat-treated at 900-1050 degrees C and an ingot is manufactured using this heat-treated furnace material made from carbon Fe and Si impurity which are contained in the furnace material made from carbon do not melt into melted copper. The content of Fe and Si impurity is 0.1 ppm, respectively. The following high grade copper ingots are obtained (b). The research findings of the commercial nozzle made from alumina and/or the commercial mold made from alumina replacing a part of above-mentioned furnace material made from carbon were obtained.

[0012] This invention is made based on these research findings (1). In the method of carrying out dissolution casting of the high grade copper raw material using the furnace material made from carbon, and manufacturing a high grade copper ingot Before carrying out dissolution casting of the high grade copper raw material, it is the furnace material made from carbon beforehand The inside of a non-oxidizing atmosphere, and temperature:800-1120 degree C (preferably) The manufacturing method of the high grade copper ingot by the furnace material made from carbon which heat-treats [900-1050-degree C] on condition of maintenance, and manufactures a high grade copper ingot using this heat-treated furnace material made from carbon, (2) It has the feature in the manufacturing method of the high grade copper ingot by the

furnace material made from carbon which replaces a part of furnace material made from carbon which the above (1) heat-treated with the nozzle made from alumina, and/or the mold made from alumina, and manufactures a high grade copper ingot.

[0013]

[Example]

As a result of preparing work examples 1-7, comparative examples 1-2 and the conventional example, and high grade electrolytic copper and analyzing this high grade electrolytic copper by GD-MS (mass spectrograph), the analysis result as shown in Table 1 was obtained.

[0014]

[Table 1]

高純度電気銅の分析結果（ppm）				
Ag	S	Si	Fe, Mg, Al, P, Ca, Cr, Ni, Zn, Sb, Te, Pb, Bi, Cd, Se, Sn, Mn	Cu
0.09	0.02	0.01	検出されず	残部

[0015] On the other hand, the commercial crucible made from high purity graphite, the nozzle made from high purity graphite, and the mold made from high purity graphite are prepared. These are heat-treated on the conditions shown in Table 2, it dissolves in 1150 degrees C and vacuum atmosphere using this heat-treated crucible made from high purity graphite, this molten metal is cast using the nozzle made from high purity graphite and mold which heat-treated [above-mentioned], and a high grade copper ingot is manufactured. Work examples 1-7 and comparative examples 1-2 were carried out.

[0016] Furthermore, the conventional example was carried out by using it, without heat-treating the crucible made from high purity graphite, the nozzle made from high purity graphite, and the mold made from high purity graphite of the above-mentioned marketing, carrying out dissolution casting of the above-mentioned high grade electrolytic copper, and manufacturing a high grade copper ingot for comparison.

[0017] The high grade copper ingot obtained in the above-mentioned work examples 1-7, comparative examples 1-2, and the conventional example was analyzed by GD-MS (mass spectrograph), and the analysis result was shown in Table 2.

[0018]

[Table 2]

種別		高純度黒鉛製ルツボ、ノズルおよび鋳型の熱処理条件			高純度銅インゴットの分析結果 (ppm)					
		温度 (℃)	雰囲気	保持時間 (hr)	Ag	S	Si	Fe	その他	Cu
実施例	1	800	Ar	2	0.09	0.02	0.04	0.04	検出されず	残
	2	850	Ar	2	0.08	0.03	0.03	0.05		
	3	900	Ar	1	0.05	0.05	0.02	0.018		
	4	950	Ar	1	0.09	0.02	0.01	0.009		
	5	1000	Ar	1	0.05	0.02	0.01	0.007		
	6	1050	Ar	1	0.08	0.02	0.01	0.01		
	7	1120	Ar	1	0.09	0.03	0.03	0.04		
比較例	1	700	Ar	5	0.09	0.09	0.2	0.8		
	2	1200	Ar	1	0.08	0.1	0.1	0.2		
従来例		-	-	-	0.09	0.2	0.9	0.8		

[0019] From the analysis result of the high-grade copper ingot shown in Table 2 to temperature: Use the crucible made from high purity graphite, nozzle, and mold which were heat-treated in 800-1120 degrees C and a non-oxidizing atmosphere. The high grade copper ingot of the manufactured work examples 1-7 is understood that there is little content of Fe and Si compared with the high grade copper ingot of the conventional example manufactured using the crucible made from high purity graphite, nozzle, and mold which are not heat-treated. Moreover, it turns out that sufficient effect is not acquired even if it heat-treats the crucible made from high purity graphite, a nozzle, and a mold at the temperature from which it separated from the range of 800-1120 degrees C, as shown in comparative examples 1 and 2.

[0020] The commercial nozzle made from high purity alumina and commercial mold other than the crucible made from high purity graphite, a nozzle, and a mold which were heat-treated on condition of the work example 4 of eight to work-example 10 table 2 are prepared, and these crucibles, a nozzle, and a mold are used. By manufacturing a high grade copper ingot from high grade electrolytic copper of Table 1, the work examples 8-10 shown in Table 3 were carried out, the obtained high grade copper ingot was analyzed by GD-MS (mass spectrograph), and the analysis result was shown in Table 3.

[0021]

[Table 3]

種別		高純度銅インゴット製造に使用したルツボ、ノズルおよび鋳型の種類			高純度銅インゴットの分析結果 (ppm)					
		ルツボ	ノズル	鋳型	Ag	S	Si	Fe	その他	Cu
実施例	8	黒鉛製	アルミナ製	アルミナ製	0.09	0.03	0.08	0.09	検出されず	残
	9	黒鉛製	アルミナ製	黒鉛製	0.09	0.02	0.09	0.07	検出されず	
	10	黒鉛製	黒鉛製	アルミナ製	0.08	0.02	0.05	0.06	検出されず	

[0022] Even if it dissolves from the work examples 8-10 of Table 3 using the crucible made from graphite heat-treated at least and others use the commercial nozzle made from high purity alumina and/or a commercial mold, Compared with the conventional example which uses the crucible made from high purity graphite, nozzle, and mold which Table 2 does not heat-treat, it turns out that Fe and a high grade copper ingot with few Si contents are obtained.

[0023]

[Effect of the Invention] As mentioned above, it is even if it uses conventionally high grade electrolytic copper which is the grade which can hardly detect Fe and Si, Although it had become the cause of the electric trouble which Fe and Si are increased and contained in the high grade copper ingot manufactured using the usual furnace material made from carbon, and Fe and Si are contained also in the wire rod obtained by rolling this ingot and carrying out wire drawing so much, and becomes various According to this invention method heat-treated before carrying out dissolution casting of the furnace material made from carbon, Fe and very few high grade copper ingots of a Si content can be manufactured, and since RRR can offer 10,000 or more wire rods inexpensive, the industrially excellent effect is brought about.

[Translation done.]